STUDY TITLE

Acute Toxicity Evaluation of Step I Pipe Discharge (WS006/WS007) from US Steel-MinnTac, Mountain Iron, Minnesota

DATA STANDARD

"Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms," 2002, Fifth Edition. EPA/821/R/02/012. U.S. Environmental Protection Agency, Washington, DC.

STUDY DATES

July 26-28, 2016

STUDY SUBMITTED

August, 2016

TESTING FACILITY

Pace Analytical 4730 Oneota Street Duluth, MN 55807

Tel. No. (218) 727-6380

PROJECT IDENTIFICATION NUMBERS

Pace Project # 1271183

NPDES Permit No. MN0057207

1.0 EXECUTIVE SUMMARY AND RESULTS

Minn Tac Step I Pipe Discharge (WS006/WS007), Mountain Iron, MN was evaluated for acute toxicity to fathead minnows (*Pimephales promelas*) during July 26-28, 2016. McNiven Creek Water (SW002) was used as the dilution and primary control water for the test. Moderately hard reconstituted water (MHRW) was used as the laboratory reference control water for the tests. The table below summarizes the toxicity results of the tests.

Fathead Minnow

48 Hour LC₅₀: >100% (95% C.I. Not Calculable) Survival NOEC: 100% Survival LOEC: --Toxicity Units (TUa): <1.00

The chemistry determinations for the final effluent samples are attached at the end of this report. The data package and statistical analysis for the test battery is also attached.

2.0 GENERAL INFORMATION

Data Standard	EPA/821/R/02/012 ·	
Testing Facility	Pace Analytical, 4730 Oneota Street, Duluth, N	AN 55807, Tel. No. (218) 727-6380
Project Director	Dan Toms	
Test Dates	July 26-28, 2016	
Report Approval	Dan Toms	Report Signature:

3.0 OBJECTIVE

To determine the median lethal concentration (LC₅₀), no- & lowest- observed effect concentrations (NOEC & LOEC) based on survival of MinnTac Step I Pipe Discharge (WS006/WS007) for fathead minnows.

4.0 REFERENCES

American Public Health Association (APHA). 1998. 20th Ed. of Standard Methods for the Examination of Water and Wastewater. APHA, Washington, D.C.

USEPA. 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fifth Edition. EPA/821/R/02/012. U.S. Environmental Protection Agency, Washington, DC.

MPCA Acute Toxicity Test Conditions for Static Renewal Whole Effluent Tests. Minnesota Pollution Control Agency.

West Inc. & University of Wyoming. TOXSTAT Software Package Version 3.5.

5.0 TEST METHODS

Test Concentrations: 0 (dilution water control), and 100%.

Test Organisms: Fathead minnow (*Pimephales promelas*) 7 days old.

Test Chambers: Fathead minnow: 800 mL glass beakers.

Incubation: Duration: 48 hours. Renewals: None

Observations: Survival was recorded each day. The test was terminated following 48 hours of exposure.

Endpoint Calculations: LC₅₀ USEPA Statistical Programs. NOEC and LOEC: TOXSTAT statistical software,

Version 3.5, West, Inc., and University of Wyoming.

6.0 DEVIATIONS FROM REFERENCED METHOD

To the best of our knowledge, no deviations from the referenced method (EPA-821-R-02-012) occurred for the study.

Cumulative Percentage of Surviving Organisms for Selected Test Species Exposed to MinnTac Step I Pipe Discharge (WS006/WS007) Tested July 26-28, 2016

Effluent Concentration (%)	Test	Interval 48 hour
	Fathead Minnow	
0 (MHRW) ^a	100° (20/20) ^d	100 (20/20)
0 (RW) ^b	100 (20/20)	100 (20/20)
100	100 (20/20)	100 (20/20)

^a Secondary Control was Moderately Hard Reconstituted Water (MHRW)

^b Primary Control Water was McNiven Creek Water (SW002)

^c Cumulative Percentage Survival

d # Alive/# Exposed



Acute Whole Effluent Toxicity Test Report

NPDES Permit Program

Doc Type: Effluent Limit Standards Review

Please read all instructions carefully before completing each section of the report. Instructions are found on Page 5.

Address the completed, signed report to:

Attn: Water Quality Submittals Minnesota Pollution Control Agency 520 Lafayette Road North St. Paul, Minnesota 55155-4194

	· · · · · · · · · · · · · · · · · · ·
Facility Information	
Permit No.: MN0057207	Station ID: WS006/WS007
Name of permittee: US Steel Minntac	
Test Information	
Test start date (mo/day/year):7/26/16	Test end date (mo/day/year): 7/28/16
or	
Check if no discharge occurred and leave the rest of the for	m blank.
Report completion date (<i>mo/day/year</i>): 8/12/16	·
WET test type (<i>check one</i>): ⊠ Initial ☐ Repeat #1 ☐ Repeat	t#2 ☐ TAC Repeat
For repeats, test start date of initial WET test (mo/day/year):	
 Dilution water (<i>check one</i>): ☐ Lab ☒ Receiving water	
f receiving water, indicate name: McNiven Creek Water (SW002	2)
Test lab name:: Pace Analytical	Phone: (218) 727-6380
Mailing address: 4730 Oneota Street	
City: Duluth State:	Minnesota Zip code: 55807
_ab representative name:Dan Toms	E-mail: dan.toms@pacelabs.com
acility representative name: Thomas Moe	F-mail: tmoe@uss.com

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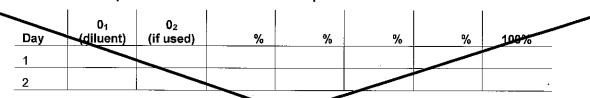
Hardness (mg/L)	56.0				992												
Alkalinity H (mg/L)				,	190												
Total Ammonia (mg/L)	× 0.20				1.6								r				
Temp °C upon test initiation	25				25										:		
Temp °C Arrival at lab	18.7				22.2				iter (mg/L)	·				f grabs:	f grabs:	f grabs:	f grabs:
TRC (mg/L)	< 0.02				< 0.02				mg/L = milligrams per Liter (mg/L)					Inter number of	inter number of	Enter number of	Enter number of
Conductivity (umhos/cm)	96.8			,	2010							e mesh size:	,	Grab-composite – Enter number of grabs:	Grab-composite - Enter number of grabs:	Grab-composite Enter number of grabs:	Grab-composite – Enter number of grabs:
PH (SU)	8.2				8.1				s per centimeter	0 N	applies)	L_] Effluent 4 If yes, state r				☐ Gra	□ Gra
First use date	7/26/16				7/26/16				umhos/cm = micromhos per centim	e) Tres No	ach one that	☐ Yes 図 No	sh sample):	⊠ Grab	☐ Grab	☐ Grab	☐ Grab
Receipt date	7/25/16				7/25/16		•		Nonmos	Does facility have chlorine limit? (check one)	\overline{c}		Effluent sample type (check one type for each sample):	24 hr composite	24 hr composite	24 hr composite	24 hr composite
Collection date	7/25/16			,	7/25/16				*If applicable	have chlorine lir	dechlorinated a	LJ Effluent 1 LJ Effluent 2 Effluent filtered? <i>(check one)</i>	ole type (check				
Sample	Receiving. Water 1	Receiving Water 2*	Receiving Water 3*	Receiving Water 4*	Effluent	Effluent 2*	Effluent 3*	Effluent 4*	*If ap	Does facility	Was sample	Effluer	Effluent samp	Effluent 1:	Effluent 2:	Effluent 3:	Effluent 4:

651-296-6300

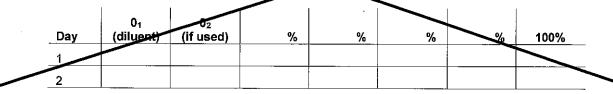
Summary of data for Fathead Minnows - Percent survival per concentration

Day	0 ₁ (diluent)	0 ₂ (if used)	%	%	%	%	100%
1	100	100			•		100
2	100	100			·	,	100
3						-	
4							

Summary of data for Ceriodaphnia dubia - Percent survival per concentration



Summary of data for Daphnia magna - Percent survival per concentration



Summary of results - Acute values

Species	LC ₅₀	TU _a (acute tòxic units)
Fathead Minnow (96 hour)	>100%	<1.00
Ceriodaphnia dubia (48 hour)		
Daphnia magna (48 hour)		

Test Acceptability Criteria (TAC)

QA/QC Criteria	Criteria met for Acute Toxicity
Fathead Minnow - Age range <24 hours at test start	☐ Yes ☒ No
Ceriodaphnia dubia - Age range <24 hours at test start	☐ Yes ☐ No
Daphnia magna – Age range <24 hours at test start	☐ Yes ☐ No
Test started within 36 hours of collection of sample	∑ Yes ☐ No
90% or greater survival of all control organisms (if any species results fail, check No)	⊠ Yes □ No

If reference toxicant other than NaCl used, please indicate:

Test deviation comments

Describe any deviation from test methods:

Per permit requirements only 48 hour fathead minnow test performed with no dilution.

(For example: pH-controlled test amount of CO₂, reduced DO levels in test leading to aeration, sample exceeded holding time.)

Certifications

Form shall be signed by the laboratory here certifying the results:

Lab representative (print):	Dap-Toms	_ Phone:	(218) 727-6380
Signature:	Dan Toms	_ Date:	8/12/16
Form shall be signed by t Applications and Reports	the permittee here in accordance with Minn.	. R. 7001.0	540 Certification of Permit
in accordance with a system submitted. Based on my ind responsible for gathering th	w that this document and all attachments were m designed to assure that qualified personnel p quiry of the person or persons who manage the ne information, the information submitted is, to am aware that there are significant penalties for sonment.	properly ga e system or the best of	ther and evaluate the information those persons directly my knowledge and belief, true,
Permittee representative (print	t): Tom Moe	_ Phone:	(218) 749-7485
Signature:		_ Date:	
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Attachments provided:			
Data package, Narrative repor	rt, Reference Toxicant Chart (Reference test met red	quirements).	

Whole Effluent Toxicity Test Instruction Sheet

This instruction sheet is provided to aid in filling out the Acute or Chronic Whole Effluent Toxicity (WET) Test report forms as required by your National Pollutant Discharge Elimination System (NPDES) Permit. If your permit requires acute WET testing, there will be one form to fill out. If your permit requires chronic WET testing, there will be two forms to fill out.

Section 1. Facility Information

Fill out your NPDES permit number, discharge outfall station identification (ID) number, and your Permittee name as it appears on your NPDES permit.

Section 2. Test Information

Test Start Date/Test End Date. Fill out the date the test began (month/day/year), the date the test finished (month/day/year)

No discharge. This box should be checked if no discharge occurred during the WET monitoring period. If no discharge occurred, the rest of the form is left blank, and should be signed, dated, and submitted by the required date.

Report Completion Date. Fill out the completion date, which is the date the report is sent from the testing laboratory as indicated on the test report (month/day/year).

WET test type. Select the appropriate form to fill out. This will be either the acute test form, or both the Fathead chronic minnow form and the Ceriodaphnia chronic form. Check the appropriate test represented by this report; either Initial, Repeat#1, Repeat#2. or TAC Repeat

Dilution water. Check either lab or receiving water, whichever was used to dilute the effluent sample to create the test series concentrations. If receiving water is used as the diluents, supply the name of that receiving water.

Test Lab Name, etc. Provide the name, phone number, address, lab representative name, and e-mail address.

Facility representative name. Provide the name of the discharging facility's representative and e-mail address.

Sample condition table. The laboratory conducting the tests must fill in the sample information and dates associated with the samples received. The information is typically collected for Chain of Custody forms and for initial sampling upon initiating a test. Provide the date the sample was collected, receipt date at the lab, date of first use for testing, pH, specific conductivity, total residual chlorine (TRC) analysis, temperature upon arrival at the lab and temperature at test initiation, total ammonia analysis, total alkalinity, and total hardness. Sample data will be provided for each sample received and as specified by the applicable acute or chronic manuals, or by the permit. Up to four samples each may be received for the receiving water and effluent for acute tests. Up to three samples may be received for chronic tests for the receiving water. Fill in any data that is below quantification levels with a less than sign and that actual quantification level (e.g. <40 ug/l) - do not report ND.

Does facility have a chlorine (TRC) permit limit? Check either Yes or No.

Was sample dechlorinated at the lab? Check each sample that was dechlorinated at the lab.

Effluent filtered? Check either Yes or No, and state the mesh size used for filtration.

Effluent sample type. For each sample collected check or check whether the sample was a twenty-four hour composite, a grab, or a grab composite with the number of grabs taken to comprise the composite sample.

Summary of data for each species for acute or chronic tests, as applicable in your permit. For each species in an acute test summarize the cumulative survival data over all replicates for each treatment level for each day of the test. Fill in the dilution series expressed as percent effluent as specified by the permit. Reaffirm which sample of either receiving water or lab water was used as the diluent (0₁) based on your answer in section 2 Test Information – Dilution water.

For a Fathead minnow chronic test summarize the cumulative 96 hour survival data over all replicates, the cumulative mean 7-day biomass data, and the 7-day cumulative mean percent survival data for each treatment level for the test. Fill in the dilution series expressed as percent effluent as specified by the permit. Reaffirm which sample of either receiving water or lab water was used as the diluent (0₁) based on your answer in section 2 Test Information – Dilution water.

For a Ceriodaphnia chronic test summarize the cumulative 48 hour survival data over all replicates, the cumulative mean 7-day reproduction/female data, and the 7-day cumulative mean percent survival data for each treatment level for the test. Fill in the dilution series expressed as percent effluent as specified by the permit. Reaffirm which sample of either receiving water or lab water was used as the diluent (0_1) based on your answer in section 2 Test Information – Dilution water.

Summary of results table for each species for acute or chronic tests, as applicable. For acute tests provide the appropriate 48 or 96 hour LC50 values for each species and the calculated toxic unit values (TUa for acute or TUc for chronic tests).

For Fathead minnow chronic tests provide the calculated IC_{25} for larval survival and growth at the end of the test as well as the critical No Observed Effect Concentration (NOEC) and Lowest Observed Effect Concentration (LOEC). Also provide the chronic toxic units (TUc) calculated as the inverse of the IC_{25} value.

For Ceriodaphnia chronic tests provide the calculated IC_{25} for survival and reproduction at the end of the test as well as the No Observed Effect Concentration (NOEC) and Lowest Observed Effect Concentration (LOEC). Also provide the chronic toxic units (TUc) calculated as the inverse of the IC_{25} value.

Test acceptability criteria. For acute tests check the corresponding boxes either yes or no for each of the Quality Assurance/ Quality Control (QA/QC) criteria.

For chronic tests check the corresponding boxes either yes or no for each of the QA/QC criteria. If there is deviation from the specified methods, please describe the change and reason for the deviation. PMSD is the percent minimum significant difference from hypothesis testing that characterizes overall test variability. The temperature variation of $\pm 1^{\circ}$ C is for individual measured test chambers over a test.

Deviations from test methods. If there is any deviation from the specified methods, please describe the change and the reason for the deviation. This is a narrative comment area to describe any unusual circumstances in conducting any of these tests.

Certifications. Provide the laboratory representative name, signature, phone number and date for the tests completed. Also provide the permittee representative name, signature, phone number and date for the tests completed. The Permittee is responsible for forwarding these results to the Minnesota Pollution Control Agency.

Attachments. Attach any narrative regarding results or SOP, bench sheets for analytical and biological data, chain of custody forms, reference test results for tested species (tables and/or charts over the past year), and toxicity calculation outputs.

www.pca.state.mn.us • 651-296-6300 • 800-657-3864 • TTY 651-282-5332 or 800-657-3864 • Available in alternative formats wq-wwprm7-40 • 4/20/10

PM: DJT

Due Date: 08/15/16

CHAIN-OF-CUSTODY / Analytical Request Docui client: USS CORP The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be c

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Document Name:

Sample Condition Upon Receipt Form

Document No.: F-DUL-C-001-Rev.01 Document Revised: 22Jan2016

Page 1 of 1

Issuing Authority: Pace Virginia, Minnesota Quality Office

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Samples Arrived with	in Hold Time?		Y.es	□No	□N/A			<u></u>
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Trip Blank Present?			∐Yes	∐No	ŪN/A			
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Project Manager Review: At two WH Date: 7-26-15

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of

hold, incorrect preservative, out of temp, incorrect containers)



Document Name:

Sample Condition Upon Receipt Form

Document No.: F-DUL-C-001-Rev.01 Document Revised: 22Jan2016

Page 1 of 1

Issuing Authority: Pace Virginia, Minnesota Quality Office

Sample Condition **Upon Receipt**

SWOOL Client Name:

Project #:

MO#: 1271183

US Steel-Min	ntac			MOH. TELTTO
Courier: ☐Fed Ex: ☐UPS ☐Commercial ►Pace	USPS Other:		lient	PM: DJT Due Date: 08/15/16 CLIENT: USS CORP
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Chain of Custody Present?	¥Yes	_□No	□N/A	1.
Chain of Custody Filled Out?	Yes	□No	□n/a	2.
Chain of Custody Relinquished?	¥Yes	□No	□N/A	3.
Sampler Name and Signature on COC?	- ₹ TYes	□No	□N/A	4.
Samples Arrived within Hold Time?	Yes	□No	 □n/a	5.
Short Hold Time Analysis (<72 hr)?	¥Yes	 □No		6. PH, CESCE
Rush Turn Around Time Requested?	□Yes	∑ No	□N/A	7
Sufficient Volume?	¥€Yes	□No		8.
Correct Containers Used?	₩Yes	□No	□N/A	9.
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Filtered Volume Received for Dissolved Tests?	□Yes	□No	∑ h/A	11. Note if sediment is visible in the dissolved containers.
Sample Labels Match COC?	Yes	□No	□N/A	12.
-Includes Date/Time/ID/Analysis Matrix: WT				
All containers needing acid/base preservation will be checked and documented in the pH logbook.	Yes	□No.	□n/a	See pH log for results and additional preservation documentation
Headspace in Methy! Mercury Container	∐Yes	□No	⋣ n/a	13.
Headspace in VOA Vials (>6mm)?	∐Yes	□No	.∏N/A	14.

THEM I MOTHICATION RESOLUTION			Field Data Required? Yes No		
Person Contacted:	•	Date/Time:	. –		
Comments/Resolution:					

□N/A

DN/A

15.

FECAL WAIVER ON FILE

Trip Blank Present?

Trip Blank Custody Seals Present?

Pace Trip Blank Lot # (if purchased):

TEMPERATURE WAIVER ON FILE Y N

Date: __ 7-26-16 Project Manager Review:

□Yes

☐Yes

□No

□No

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of hold, incorrect preservative, out of temp, incorrect containers)

MinnTac

Pace Analytical

Client:	MinnTac
Pace Pr	oject #: 1271183
Test:	Acute Toxicity Evaluation
Test Init	iation Date: July 26, 2016
Test Ter	mination Date: July 28, 2016

ENVIRONMENTAL SAMPLE TEST INFORMATION

Date: July 26, 2016
Client: MINNTAC
Pace Project #: 1271183
Dilution Water: SW002
Test Chamber: 800 mL glass
Food: None
Temperature: 25°C
Test Organism(s)/Age: Fathead Minnow/7 day
Comments:
· · · · · · · · · · · · · · · · · · ·
· · · · · · · · · · · · · · · · · · ·

TOXICITY TEST RENEWAL FORM

CLIENT: MINNTAC	PACE PROJECT #: 1271183
TEST: Acute Toxicity Evaluation	TEST INITIATION DATE: July 26, 2016
ORGANISM: Fathead Minnow	TERMINATION DATE: July 28, 2016

TEST DAY	0 Test Initiation	1	2	3	4
DATE	7/20/16	7/27/16	7/28/16		
]
TIME OF READING					
	1456	14.21	FHM 14 17	FHM	FHM
TIME OF FEEDING					
DILUTION WATER	RW 1271183.00℃	RW 1271183 co2	RW 1271183- 002	RW	RW
	MHRW 16-04D	MHRW 16-040	MHRW 16 -040	MHRW .	MHHW
INITIALS	de	æ	CJA		1

INITIAL CHEMISTRIES

CLIENT: MINNTAC	Pace Project #:	1271183				
TEST: Acute Toxicity E	valuation TEST INITIAT	TINITIATION DATE: July 26, 2016				
ORGANISM(S): Fathea	d Minnow • TEST TERMIN	NATION DATE:	July 28, 2016			
	7126/16 CJA /					
CONCENTRATION:	MHRW					
pH (su)	8-15					
DO (mg/l)	8.0					
Cond (umhos/cm)	318					
CONCENTRATION:	SW002 Receiving Water			*		
pH (su)	7.27	<u> </u>	1			
DO (mg/l)	7.9					
Cond (umhos/cm)	100+ CJA 96,7					
CONCENTRATION:	100% (WS006/007) 100% Effluent	t				
pH (su)	8.17	<u> </u>	<u> </u>			
DO (mg/l)	7.9	1				
Cond (umhos/cm)	2070					
Daily T	Temperatures					
2	24.5					
·	FINAL CHEMISTI	RIES				
	7127116 CLC	7128116	6 / CZ	A		
CONCENTRATION:	ta san ka walan wasan a san a fara a san a s					
pH (su)	7.92	γ.,	<u>.v</u>	<u> </u>		

	7127116 1 CLC	7128116 / CJA
CONCENTRATION:	MHRW	
pH (su)	7,92	8.0
DO (mg/l)	8,3	8.0
CONCENTRATION:	SW002 Receiving Water	
pH (su)	7.32	7.66
DO (mg/l)	7.9	7.9
CONCENTRATION:	100% (WS006/007) 100% Effluent	
pH (su)	8118	8.35
DO (mg/l)	811	8.0

Daily Temperatures

Bath# 2 24.6

ACUTE TOXICITY DATA LOG

Client: MINNTAC
Project #: 1271183
Test: Acute Toxicity Evaluation
Test Initiation Date: July 26, 2016
Investigator: Toms
Test Duration: 48 hour
Renewal: None / Daily / Other

Species: Fathead Minnow

Age: 10 day

No. Animals/No. Reps: 10/2

Sources of Animals: ABS

Dilution Water/Control: River Water

Test Volume: 600 mL

Test Temperature: 25°C

Minimum Control Survival ≥ 90%:(∑/N)

Concentration	Survival Readings; (# alive out of # exposed from above unless shown otherwise)					
		Hour licate B		Hour plicate B	72 Hour Replicate A B	96 Hou Replicate A B
MHRW	10	10	10	10		
SW002 (RW)	10	10	10	10		
WS006/007 (100% Eff)	16	10	10	10		
	Dated Initials	CJA	Dated Initials 7128116	` cJA	Dated Initials	Dated Initials
Comments:			•			
		<u> </u>	····			